



Processing flexible glass Thin film stress and its influence on glass durability

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26/04/2023, Session 7: Reliability of organic electronics

Glass with thicknesses below 100 µm is flexible



Flexible glass has outstanding characteristics

🕖 Fraunhofer



OLED lighting



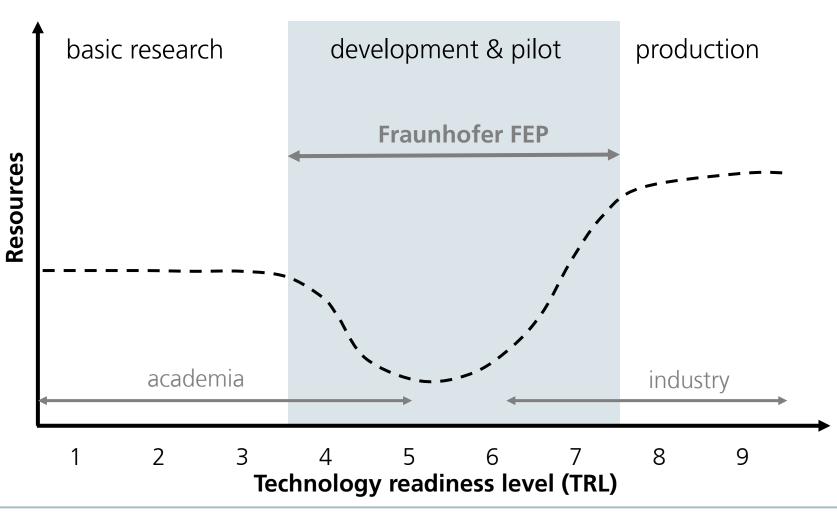




Smartphone optics



...but it is partly stuck in the technological valley of death



- Only a few flexible glass applications already reached TRL 8-9
- Mechanical reliability during production is one of the main challenges
- Research focus at FEP: TRL 4-7



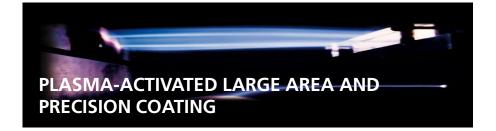
In imitation of REHVA Journal 03/2015 p.58

Core Competencies of Fraunhofer FEP















Material properties of flexible glass along the process chain

Publicly funded project CUSTOM

- Identification of the most crucial process steps to prevent glass fracture due to limited edge strength or glass durability
- Correlation of cutting techniques, coating process parameters etc. with fracture strength and fatigue behavior
- Results can be used to define parameter fields for reliable processing
 - suitable role diameters for winding
 - adequate contact pressure during handling
- Industrial advisory committee consists of 14 partners along the whole process chain



Material properties of flexible glass along the process chain Advisory committee



Vacuum inline coating machine with 10 process stations



DC and PMS PVD and PECVD planar and rotary targets



max. 600 x 1200 mm²



pre- and post annealing Inline flash lamp annealing





ITO coatings with different thin film stress values

150 nm ITO

100 µm flexible glass

Deposition	room temperature	Furnace annealing (300 °C, 15 min, air)
Specific resistance	345 μΩm	255 μΩm
Thin film stress	-700 MPa	- 1100 MPa

Two selected samples out of approx. 35 in the project

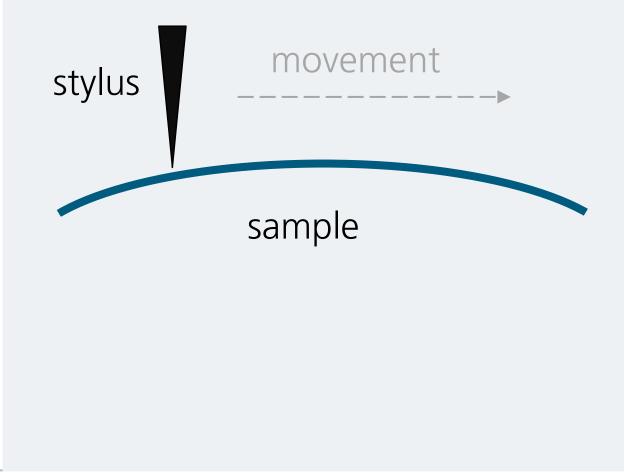


Thin film stress measurement

Profilometry using STONEY equation

$$\sigma = \frac{1}{6} \cdot \left(\frac{1}{R_{post}} - \frac{1}{R_{pre}} \right) \cdot \frac{E}{1 - \nu} \cdot \frac{d_s^2}{d_f}$$

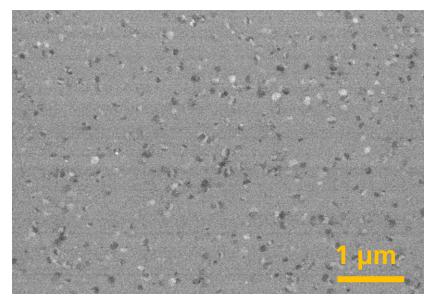
σ	Thin film stress
R _{pre/post}	Bending radius before and after thin film deposition
<i>E</i>	Young's modulus of the substrate
ν	Poisson ratio of the substrate
d _s	Substrate thickness
$d_{f} \ldots$	Thin film thickness

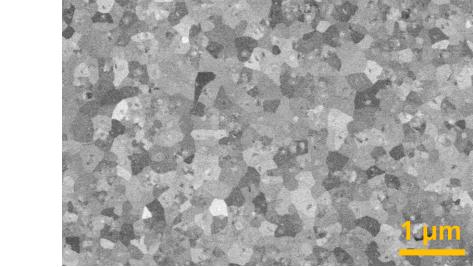




Annealing of ITO leads to full crystallization

as deposited (-700 MPa)





air annealed (-1100 MPa)

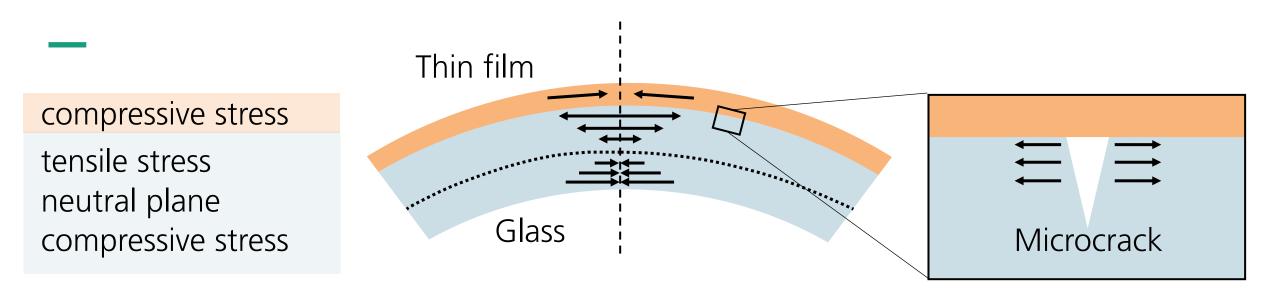
- Increased stress after annealing because of oxygen incorporation
- Relatively high stress values set to determine a clear influence on glass strength

 $+0_{2}$

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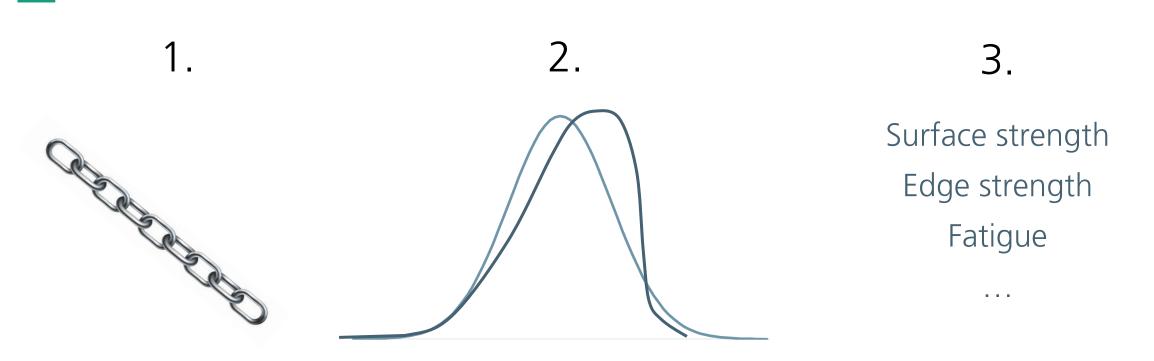
Tensile stress in the glass substrate leads to crack propagation



- Compressive stress in the thin film leads to a stress profile in the glass with tensile stress in the near-surface region
- Tensile stress in the glass substrate promotes subcritical crack growth i.e. it enables microcracks to propagate



Everything you need to know about glass strength (today)



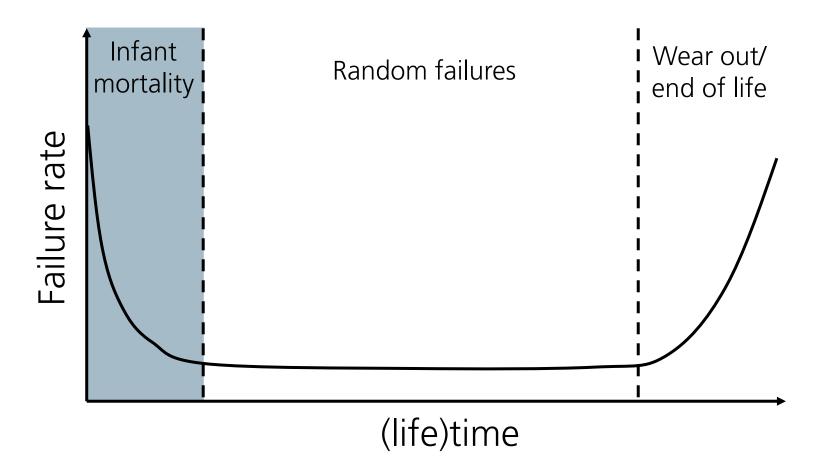
Failure at the weakest point, often the edge

Randomly distributet, following Weibull distribution

Product characteristic, not a material characteristic



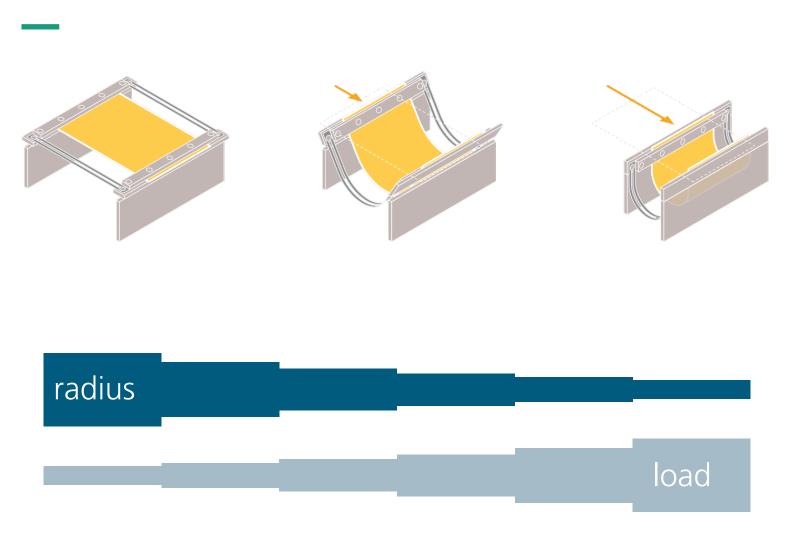
The failure rate of flexible glass during functionalization is high



- Infant mortality is highly relevant in the flexible glass process chain
- Lower infant mortality will enable viable production yields
- → Research focus on early life fatigue, i.e. several 1000 load cycles

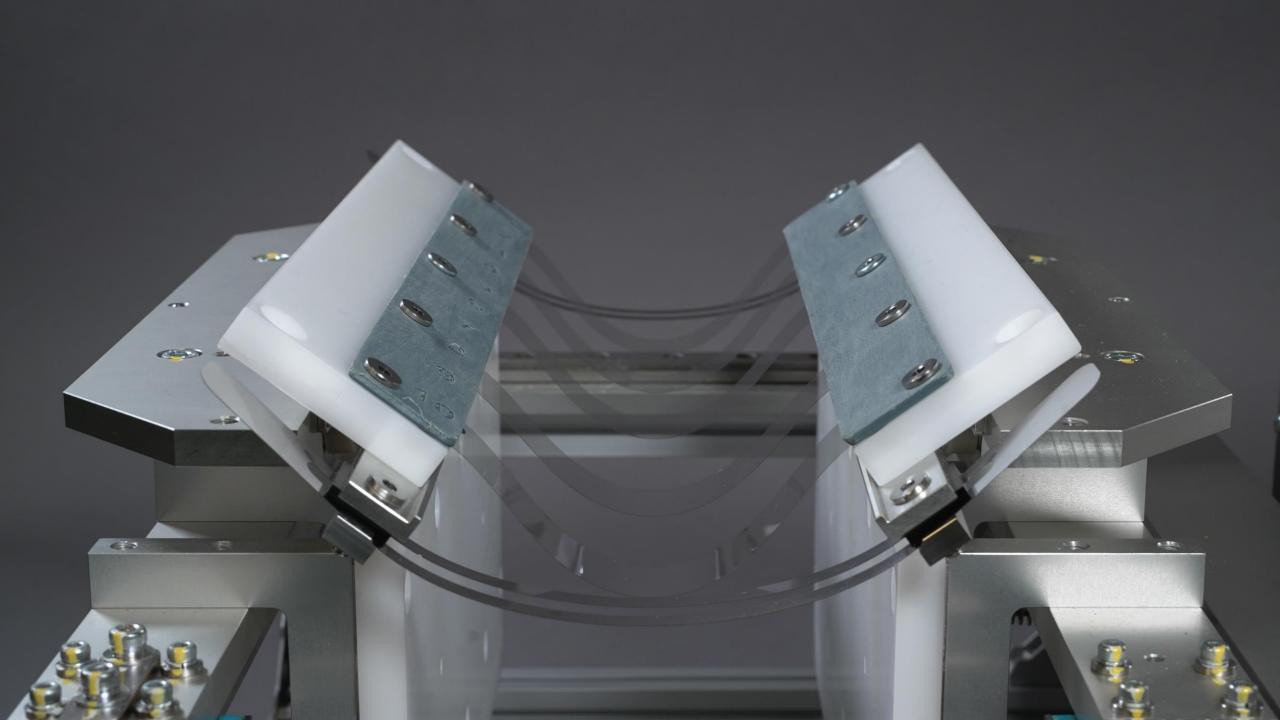


Stepwise testing to find the zone of fatigue failure



- Butterfly folding motion
- Tension-Free U-shape Folding Tester (Bayflex/ Yuasa)
- newly developed specimen holder for thin glass
- 500 bending cycles per radius, then load increase
- sample size: 30 specimens
- Test range: 130 500 MPa in steps of 25 MPa

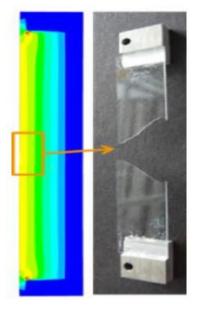


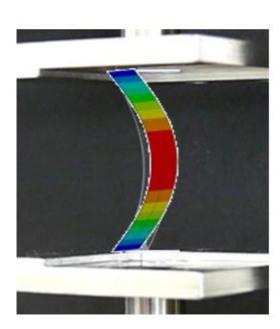


Each edge strength test generates a different stress state

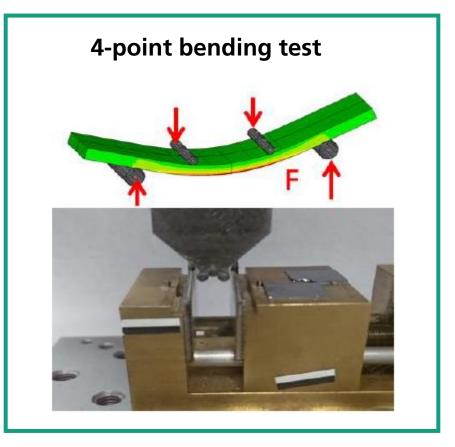
asymmetric tensile test

Intellectual property of Schott AG Patent No: DE 10 2014 110 856 B4





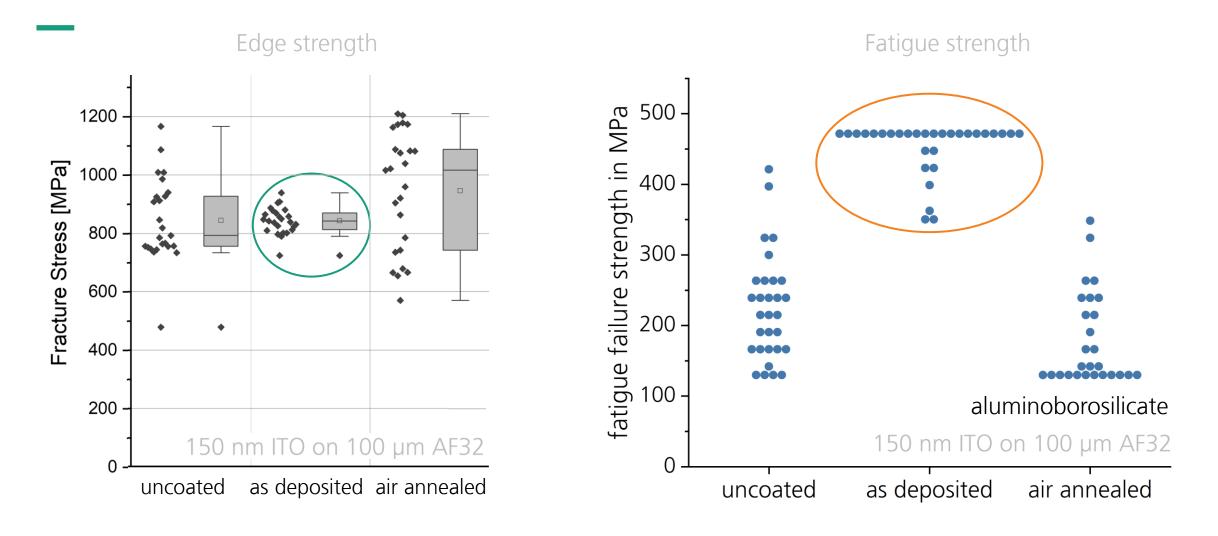
2-point bending test



- Edge strength was determined using different tests
- Here: focus on results of the 4-point bending test

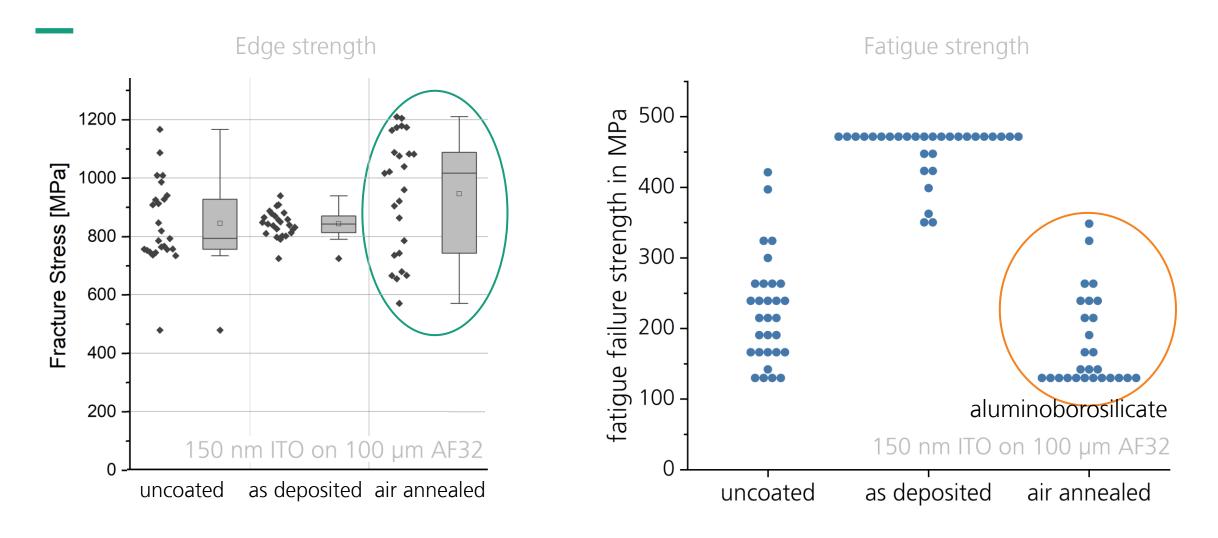


ITO coating leads to a significant (fatigue) strength increase



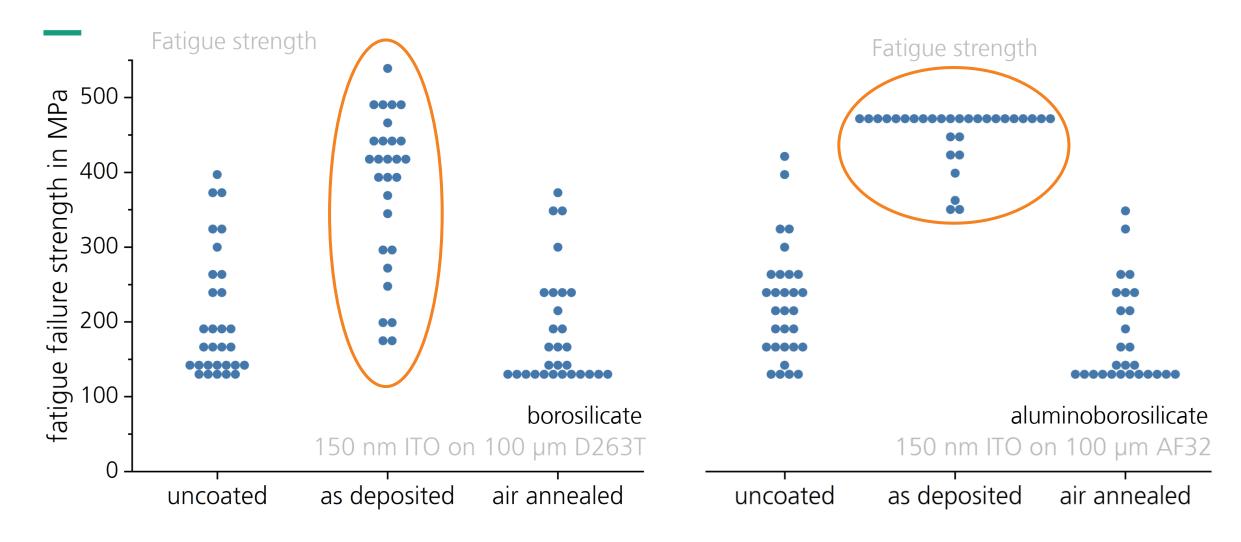


Air annealing leads to opposing effects in the two test set-ups



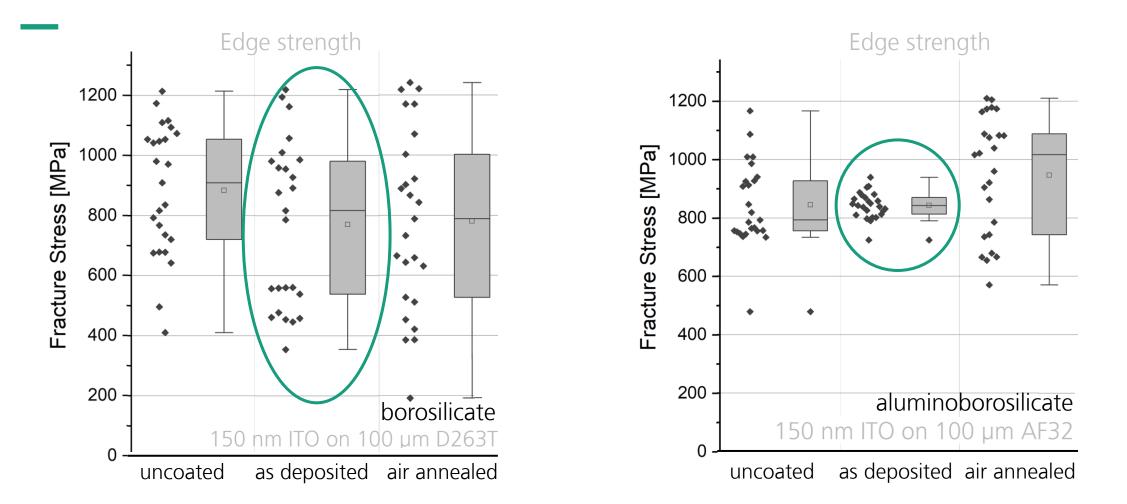


The statistical distribution differs for different glass types





...in both test setups





Strategies for reliable flexible glass handling

Key results

- Thin film stress influences the strength of coated flexible glass
- Coating can lead to a strength increase or decrease
- Different test methods show different results

Stress management

- Increasing importance with decreasing substrate thickness
- Crucial for process stability

Alternative annealing methods

- Vacuum annealing
- Deposition at elevated temperature levels
- Flash Lamp Annealing

Improved understanding of glass strength

- Mitigate risks during handling and functionalization
- Allow reliable processing with high yield











Federal Ministry for Economic Affairs and Climate Action

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